FORM PTO-1390 (Modified) (REV 11-98) U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER	U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR
205738US0PCT	09/806473

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/IB99/01625	01 October 1999	07 October 1998

TITLE OF INVENTION
NOVEL RHEOLOGY REGULATORS OF THE CRUSHED NATUAL CALCIUM CARBONATE TYPE, POSSIBLY TREATED WITH A FATTY ACID OR ITS SALT, AND THEIR APPLICATION IN POLYMERIC COMPOSITIONS

APPLICANT(S) FOR DO/EO/US
Pierre BLANCHARD, et al.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371 (c) (2))
   a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☒ has been transmitted by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ A copy of the International Search Report (PCT/ISA/210).
8. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371 (c)(3))
   a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
   b. ☐ have been transmitted by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
9. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
10. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371 (c)(4)).
11. ☐ A copy of the International Preliminary Examination Report (PCT/IPEA/409).
12. ☐ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371 (c)(5)).

Items 13 to 20 below concern document(s) or information included:

13. ☐ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
14. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
15. ☐ A **FIRST** preliminary amendment.
16. ☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
17. ☐ A substitute specification.
18. ☐ A change of power of attorney and/or address letter.
19. ☐ Certificate of Mailing by Express Mail
20. ☒ Other items or information:

Request for Consideration of Documents Cited in International Search Report
Notice of Priority

U.S. APPLICATION NO. (IF KNOWN, SEE 37 CFR)	INTERNATIONAL APPLICATION NO.	ATTORNEY'S DOCKET NUMBER
09/806473	PCT/IB99/01625	205738US0PCT

21. The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)) :

		CALCULATIONS PTO USE ONLY
☐	Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2) paid to USPTO and International Search Report not prepared by the EPO or JPO $1,000.00	
☒	International preliminary examination fee (37 CFR 1.482) not paid to USPTO but Internation Search Report prepared by the EPO or JPO $860.00	
☐	International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO $710.00	
☐	International preliminary examination fee paid to USPTO (37 CFR 1.482) but all claims did not satisfy provisions of PCT Article 33(1)-(4) $690.00	
☐	International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) $100.00	
	ENTER APPROPRIATE BASIC FEE AMOUNT =	$860.00
	Surcharge of **$130.00** for furnishing the oath or declaration later than ☐ 20 ☒ 30 months from the earliest claimed priority date (37 CFR 1.492 (e)).	$130.00

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total claims	- 20 =	0	x $18.00	$0.00	
Independent claims	- 3 =	0	x $80.00	$0.00	
Multiple Dependent Claims **(check if applicable)**.			☐	$0.00	
	TOTAL OF ABOVE CALCULATIONS =			$990.00	
	Reduction of 1/2 for filing by small entity, if applicable. Verified Small Entity Statement must also be filed (Note 37 CFR 1.9, 1.27, 1.28) **(check if applicable)**. ☐			$0.00	
	SUBTOTAL =			$990.00	
	Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492 (f)). +			$0.00	
	TOTAL NATIONAL FEE =			$990.00	
	Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31) **(check if applicable).** ☐			$0.00	
	TOTAL FEES ENCLOSED =			$990.00	
				Amount to be: refunded	$
				charged	$

☒ A check in the amount of **990.00** to cover the above fees is enclosed.

☐ Please charge my Deposit Account No. in the amount of to cover the above fees.
A duplicate copy of this sheet is enclosed.

☒ The Commissioner is hereby authorized to charge any fees which may be required, or credit any overpayment to Deposit Account No. **15-0030** A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

22850

Surinder Sachar
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Surinder Sachar
SIGNATURE

Norman F. Oblon
NAME

24,618
REGISTRATION NUMBER

April 9 2001
DATE

205738US0 PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF: :

PIERRE BLANCHARD ET AL : ATTN: NEW APPLICATION DIVISION

SERIAL NO: 09/806,473 :

FILED: April 9, 2001 :

FOR: NOVEL RHEOLOGY REGULATORS SUCH AS GROUND NATURAL CALCIUM CARBONATES OPTIONALLY TREATED WITH A FATTY ACID OR SALT AND THEIR USE :

PRELIMINARY AMENDMENT

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Prior to examination on the merits, please amend the above-identified application as follows:

IN THE CLAIMS

Please amend the claims as shown in the marked-up copy to read as follows:

7. (Amended) A rheology regulator according to Claim 1, characterised in that it is a case of a natural calcium carbonate treated by means of at least one fatty acid containing 10 to 24 atoms of carbon or its salt chosen from amongst the salts of calcium, magnesium, zinc or a mixture thereof and more particularly using stearic acid or its calcium salt in a proportion of around 0.01% to 5% by weight.

9. (Amended) A rheology regulator according to Claim 1, characterised in that it has an oil absorption which is greater than 16 measured according to ISO 787-V (Rub-out method).

10. (Amended) Use of a rheology regulator according to Claim 1, for the preparation of sealants, adhesives or plastisols.

11. (Amended) Use of a rheology regulator according to Claim 1 for the preparation of rubbers.

12. (Amended) Use as a rheology regulator, of dispersions or suspensions, in an organic medium, of a natural calcium carbonate crushed to a high degree of fineness according to Claim 1 for the preparation of sealants or coatings, adhesives or plastisols.

13. (Amended) Use as a rheology regulator, of dispersions or suspensions, in an organic medium, of a natural calcium carbonate crushed to a high degree of fineness according to Claim 1, for the preparation of rubbers.

14. (Amended) A plastisol, characterised in that it comprises a rheology regulator according to Claim 1.

15. (Amended) A rubber, characterised in that it comprises a rheology regulator according to Claim 1.

16. (Amended) A sealant or coating or adhesive, characterised in that it comprises a rheology regulator according to Claim 1.

18. (Amended) A sealant or coating or adhesive according to Claim 1, characterised in that it comprises in addition to one or more additives chosen from amongst smoked silica as a thixotropic agent, a bleaching agent such as TiO_2, UV stabilizers, adhesion promoter, a catalyst such as dibutylin dilaurate and dehydrating agents such as a silane.

REMARKS

Claims 1-18 are active in the present application. The claims are amended to remove multiple dependencies. No new matter is added. An action on the merits and allowance of the claims is solicited.

Respectfully submitted,

OBLON, SPIVAK, McCLELLAND,
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205738US0 PCT

Marked-Up Copy
Serial No: 09/806,473
Amendment Filed on:
08/01/01

IN THE CLAIMS

Please amend the claims as follows:

--7. (Amended) A rheology regulator according to [any one of Claims 1 to 6] Claim 1, characterised in that it is a case of a natural calcium carbonate treated by means of at least one fatty acid containing 10 to 24 atoms of carbon or its salt chosen from amongst the salts of calcium, magnesium, zinc or a mixture thereof and more particularly using stearic acid or its calcium salt in a proportion of around 0.01% to 5% by weight.

9. (Amended) A rheology regulator according to [any one of Claims 1 to 8] Claim 1, characterised in that it has an oil absorption which is greater than 16 measured according to ISO 787-V (Rub-out method).

10. (Amended) Use of a rheology regulator according to [any one of Claims 1 to 9] Claim 1, for the preparation of sealants, adhesives or plastisols.

11. (Amended) Use of a rheology regulator according to [any one of Claims 1 to 9] Claim 1 for the preparation of rubbers.

12. (Amended) Use as a rheology regulator, of dispersions or suspensions, in an organic medium, of a natural calcium carbonate crushed to a high degree of fineness according to [any one of Claims 1 to 9] Claim 1 for the preparation of sealants or coatings, adhesives or plastisols.

13. (Amended) Use as a rheology regulator, of dispersions or suspensions, in an

organic medium, of a natural calcium carbonate crushed to a high degree of fineness according to [any one of Claims 1 to 9] Claim 1, for the preparation of rubbers.

14. (Amended) A plastisol, characterised in that it comprises a rheology regulator according to [any one of Claims 1 to 9] Claim 1.

15. (Amended) A rubber, characterised in that it comprises a rheology regulator according to [any one of Claims 1 to 9] Claim 1.

16. (Amended) A sealant or coating or adhesive, characterised in that it comprises a rheology regulator according to [any one of Claims 1 to 9] Claim 1.

18. (Amended) A sealant or coating or adhesive according to [either one of Claims 16 and 17] Claim 1, characterised in that it comprises in addition to one or more additives chosen from amongst smoked silica as a thixotropic agent, a bleaching agent such as TiO_2, UV stabilizers, adhesion promoter, a catalyst such as dibutylin dilaurate and dehydrating agents such as a silane.--

DOCKET NO. 205738US0PCT

IN RE APPLICATION OF: BLANCHARD Pierre et al.

SERIAL NO.: 09/806,473

FILED: 09 April 2001

FOR: NOVEL RHEOLOGY REGULATORS SUCH AS GROUND NATURAL CALCIUM CARBONATES OPTIONALLY TREATED WITH A FATTY ACID OR SALT AND THEIR USE

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

Sir:

Transmitted herewith is an amendment in the above-identified application.

☐ No additional fee is required.

☐ Small entity status of this application under 37 C.F.R. §1.9 and §1.27 has been established by a verified statement previously submitted.

☐ Small entity status of this application under 37 C.F.R. §1.9 and §1.27 has been established by a verified statement submitted herewith.

☒ Additional documents filed herewith: Response to Notification/Notification of Missing Requirements
Preliminary Amendment/Declaration/Petition for Extension

The fee has been calculated as shown below.

	(Col. 1)		(Col. 2)	(Col. 3)	SMALL ENTITY		OTHER THAN A SMALL ENTITY	
	CLAIMS REMAINING AFTER		HIGHEST NUMBER PREVIOUSLY PAID FOR	PRESENT EXTRA	RATE	ADDITIONAL FEE	RATE	ADDITIONAL FEE
TOTAL	* 18	MINUS	** 20	=	X9 =	$	X18 =	$0.00
INDEP	* 1	MINUS	*** 3	=	X40 =	$	X80 =	$0.00
☐	FIRST PRESENTATION OF MULTIPLE DEPENDENT CLAIM				+135=	$	+270=	$0.00
					TOTAL	$	TOTAL	$0.00

__ A check in the amount of $_____ is attached.

XX Please charge any additional fees for the papers being filed herewith and for which no check is enclosed herewith, or credit any overpayment to deposit Account No. 15-0030. A duplicate copy of this sheet is enclosed.

XX If these papers are not considered timely filed by the Patent and Trademark Office, then a petition is hereby made under 37 C.F.R. §1.136, and any additional fees required under 37 C.F.R. §1.136 for any necessary extension of time may be charged to deposit Account No. 15-0030. A duplicate copy of this sheet is enclosed.

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*If the entry in Column 2 is less than the entry in Column 1 write "0" in Column 3.
**If the "Highest Number Previously paid for" IN THIS SPACE is less than 20 write "20" in this space.
***If the "Highest Number Previously paid for" IN THIS SPACE is less than 3 write "3" in this space.7/93

09/806473

JC08 Rec'd PCT/PTO 09 APR 2001

NOVEL RHEOLOGY REGULATORS OF THE CRUSHED NATURAL CALCIUM CARBONATE TYPE, POSSIBLY TREATED WITH A FATTY ACID OR ITS SALT, AND THEIR APPLICATION IN POLYMERIC COMPOSITIONS

The present invention relates to the technical sector of sealants, coatings, adhesives, plastisols or rubbers.

There are known, in this field, polymeric compositions, with or without fillers, for example polyurethanes with a silane termination which are used as sealants or adhesives setting in moist conditions.

In the presence of moisture, terminal silane groups undergo, in a known manner, a hydrolysis and condensation reaction. A stable siloxane lattice (Si-O-Si) then forms.

Such products have many applications in various industrial fields such as the transport and building industries.

More and more sophisticated formulations have therefore been sought, notably of the "single-component" type, capable of being applied to various substrates posing problems which are more and more difficult to resolve.

The composition of this type of formulation comprises one or more fillers, which can be one or more calcium carbonates normally referred to as "fine".

It has been discovered according to the invention that, surprisingly, the selection of a natural calcium carbonate ground to a high degree of fineness, which will be described below, or this calcium carbonate treated also as described below, makes it possible to achieve an unequalled set of satisfactory properties for the final product. These include notably good adaptability to adhesion on many types of substrate, such as certain difficult plastics, including without a prior primer coating called "primer", a reduction in the quantity of polymer required (and therefore a reduction in the cost of materials), or an appreciable reduction in the mixing time (which may reach a factor of 1/2 for each step, which has an obvious great economic advantage).

It is even more surprising to find that this remarkable improvement in a set of properties of the final product does not take place, as an expert would logically

predict, to the detriment of the final mechanical properties, or properties such as resistance to chemical agents or UV radiation or similar properties conventionally required of such agents.

Formulations of the plastisol type based on polyvinyl chloride (PVC) are also known.

It should be stated here that a plastisol designates a suspension of one or more PVC resins in a liquid plasticiser and additives such as mineral fillers, stabilisers, mineral and/or organic pigments, expansion agents, adhesion promoters, fluidifiers and others.

After thermal gelling, the plastisol takes the appearance of a more or less flexible compact mass.

One of the mineral fillers normally used consists of a synthetic calcium carbonate obtained chemically (precipitated calcium carbonate: PCC) such as for example the product Winnofil SPT Premium™ from Zeneca.

According to the invention, it is proposed to use, as a rheology regulating mineral material, a natural calcium carbonate crushed to a high degree of fineness whether or not with a dispersing agent present. This natural calcium carbonate is chosen from amongst chalk, calcite or marble, alone or in a mixture, or from amongst these same calcium carbonates treated by means of at least one fatty acid or its salt or a mixture thereof and preferentially using stearic acid or its salt, such as notably calcium, magnesium or zinc stearate and highly preferably using stearic acid or its calcium salt, the whole as described in more detail below.

The rheology regulator product according to the invention is characterised in that it is a case of a natural calcium carbonate, with a specific surface area of around 14 to 30 m^2/g, preferably 16 to 24 m^2/g and highly preferentially around 20 m^2/g, measured according to the BET method to ISO 4652.

This carbonate is possibly treated with at least one fatty acid or its salt or a mixture thereof, an acid which is an acid containing 10 to 24 atoms of carbon, and more particularly stearic acid or its salt, such as notably calcium, magnesium or zinc stearate and highly preferentially by means of stearic acid or its calcium salt,

preferably at the rate of 0.01% to 5% by weight and more preferentially 1% to 4% by weight.

It should be noted here that stearic acid means stearic acids of industrial quality composed mainly of 50% to 70% octadecanoic acid and 30% to 50% hexadecanoic acid.

An original advantage of the product according to the invention is that this product is a calcium carbonate which fulfils a rheology regulating function. This function is normally devolved to the polymers and additives contained in the polymeric formulation, such as for example viscosity depressors, and the Applicant was surprised to see it fulfilled by a product of the type consisting of a material with a natural mineral filler with a high degree of fineness.

The product selected according to the invention consists of a natural calcium carbonate crushed very finely with a dispersant present or not and possibly treated by means of at least one fatty acid or its salt or a mixture thereof.

Another of its characteristics lies in its oil absorption, which is greater than 16, measured according to ISO 787-V (Rub-out method).

A calcium carbonate with a specific surface area of 19 to 26 m^2/g was described in the patent EP 0 795 588. It is known according to this document as a pigment giving brightness and opacity in the papermaking field. This field of application is totally different. In addition, such a function is completely different from that of a rheology regulating action, and nothing would suggest either the function brought to light by the invention, nor the fact that this novel application could lead to a surprising set of properties having a great economic advantage.

The invention also relates to the use, as a rheology regulator for the preparation of sealants or coatings, adhesives, plastisols or rubbers, of these natural calcium carbonates crushed to a very high degree of fineness, possibly treated by means of at least one fatty acid or its salt, notably of calcium, magnesium or zinc or a mixture thereof. This acid, which is an acid containing 10 to 24 atoms of carbon, is more particularly stearic acid or its salt, such as notably calcium, magnesium or zinc stearate and highly preferentially stearic acid or its calcium salt. The processing takes

place preferably at the rate of 0.01% to 5% by weight and more preferentially 1% to 4% by weight of at least one fatty acid or its salt or a mixture thereof.

The invention also relates to the use of dispersions or suspensions, in an organic medium, of these calcium carbonates, treated or non-treated, as a rheology regulator for the preparation of sealants or coatings, adhesives, plastisols or rubbers.

The invention also relates to polymeric compositions of plastisols, sealant or coating, elastomer or rubber containing, as a rheology regulator, the said natural calcium carbonates crushed to a high degree of fineness, possibly treated by means of at least one fatty acid or its salt or a mixture thereof.

Other characteristics and advantages of the invention will be understood more clearly from a reading of the following description and examples. These examples should not be taken to represent any limitative aspect of the invention.

In the examples, the products have the following characteristics:

PRODUCT A:

A filler of the prior art, consisting of a natural calcium carbonate of the chalk type, crushed and dried, not treated, with a mean grain diameter of 0.67 micrometres measured by means of the Sedigraph 5100 from Micromeritics, with a specific surface area of 11 m^2/g measured according to the BET method to ISO 4652.

PRODUCT B:

A rheology regulator according to the invention, consisting of a natural calcium carbonate of the chalk type, crushed and dried, not treated, with a mean grain diameter of 0.60 micrometres measured by means of the Sedigraph 5100 from Micromeritics, with a specific surface area of 19.5 m^2/g measured according to the BET method to ISO 4652 and with an oil absorption of 18.75 measured according to ISO 787-V (Rub-out method).

PRODUCT C:

A rheology regulator according to the invention, consisting of a natural calcium carbonate of the Urgonian calcite type, crushed by the wet method and dried,

not treated, with a mean grain diameter of 0.44 micrometres measured by means of the Sedigraph 5100 from Micromeritics, with a specific surface area of 16.5 m^2/g measured according to the BET method to ISO 4652 and with an oil absorption of 20 measured according to ISO 787-V (Rub-out method).

PRODUCT D:

A rheology regulator according to the invention, consisting of a natural calcium carbonate of the chalk type, crushed by the wet method and dried, treated by the use of 3% by weight stearic acid, with a mean grain diameter of 0.59 micrometres measured by means of the Sedigraph 5100 from Micromeritics, with a specific surface area, after treatment, of 16 m^2/g measured according to the BET method to ISO 4652 and with an oil absorption, after treatment, of 16.3 measured according to ISO 787-V (Rub-out method).

PRODUCT E:

A rheology regulator according to the invention, consisting of a natural calcium carbonate of the Urgonian calcite type, crushed by the wet method and dried, not treated, with a mean grain diameter of 0.58 micrometres measured by means of the Sedigraph 5100 from Micromeritics, with a specific surface area of 14.4 m^2/g measured according to the BET method to ISO 4652 and with an oil absorption of 17.9 measured according to ISO 787-V (Rub-out method).

PRODUCT F:

A rheology regulator according to the invention, consisting of a natural calcium carbonate of the chalk type, crushed by the wet method and dried, treated by the use of 1% by weight stearic acid, with a resulting granulometry of 96% < 1 micrometre and 39% < 0.2 micrometres measured by means of the Sedigraph 5100 from Micromeritics, with a specific surface area, after treatment, of 28 m^2/g measured according to the BET method to ISO 4652 and with an oil absorption, after treatment, of 19.5 measured according to ISO 787-V (Rub-out method).

PRODUCT G:

A rheology regulator according to the invention, consisting of a natural calcium carbonate of the chalk type, crushed by the wet method and dried, not treated, with a specific surface area of 22 m^2/g measured according to the BET method to ISO 4652 and an oil absorption of 19.4 measured according to ISO 787-V (Rub-out method).

PRODUCT H:

A filler of the prior art consisting of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™.

PRODUCT I:

A filler of the prior art, consisting of natural calcium carbonate of the chalk type, crushed by the wet method and dried, treated by means of 1% stearic acid, with a mean diameter of 1.4 micrometres measured by means of the Sedigraph 5100 from Micromeritics and with a specific surface area of 6 m^2/g measured according to the BET method to ISO 4652.

PRODUCT J:

A filler of the prior art, consisting of a natural calcium carbonate of the chalk type, crushed by the wet method and dried, treated by the use of 1% by weight stearic acid, with a mean diameter of 1 micrometre measured by means of the Sedigraph 5100 from Micromeritics and a specific surface area of 10 m^2/g measured according to the BET method to ISO 4652.

PRODUCT K:

A filler of the prior art consisting of a treated precipitated calcium carbonate sold by Solvay under the name Socal U1S2.

EXAMPLE 1:

This example relates to the use of calcium carbonate as a rheology regulator for the preparation of plastisols.

In these tests, tests were carried out on the replacement of the synthetic calcium carbonate obtained by precipitation, of the prior art, or precipitated calcium carbonate (PCC), by the specific natural calcium carbonate according to the invention.

In a formulation of the plastisol type based on PVC (polyvinyl chloride) containing no calcium carbonate, it was sought to compare the effect of the replacement of 50% to 100% of the mineral filler normally used, namely a precipitated calcium carbonate, by a natural calcium carbonate crushed to a high degree of fineness according to the invention.

To do this, with 75 g of plastisol without filler, the calcium carbonate to be tested was mixed in a 7 cm diameter receptacle and the mixture was homogenised with a spatula. Then the mixture was put in dispersion for two minutes using a "Pendraulik"™ LD50 laboratory mixing appliance, the diameter of the dispersing disc being 5 cm, the speed of rotation of the disc being 2700 rev/min (manual setting at position 3).

The dispersing being terminated, the viscosity was measured by means of "Rheomat 120"™ equipment, a measuring appliance according to DIN 125, at 20°C.

Test N° 1:

This test illustrates the prior art and uses 20 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H) and 5 g of natural calcium carbonate sold under the name Juraperle™ BS by Juraweiss.

Test N° 2:

This test illustrates the prior art and uses 13 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H) and 12 g of product A according to the prior art.

Test N° 3:

This test illustrates the invention and uses 25 g of product B according to the invention.

Test N° 4:

This test illustrates the prior art and uses 20 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H).

Test N° 5:

This test illustrates the invention and uses 10 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H) and 15 g of product D according to the invention.

Test N° 6:

This test illustrates the invention and uses 10 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H) and 15 g of product C according to the invention.

Test N° 7:

This test illustrates the invention and uses 13 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H) and 12 g of product E according to the invention.

Test N° 8:

This test illustrates the invention and uses 10 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H), 5 g of natural calcium carbonate sold under the name Juraperle™ BS by Juraweiss and 10 g of product D according to the invention.

Test N° 9:

This test illustrates the invention and uses 10 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H), 5 g of natural

calcium carbonate sold under the name Juraperle™ BS by Juraweiss and 10 g of product E according to the invention.

Test N° 10:

This test illustrates the invention and uses, for a mixture with 72 g of plastisol, 10 g of a precipitated calcium carbonate sold by Zeneca under the name Winnofil SPT™ (product H), 5 g of natural calcium carbonate sold under the name Juraperle™ BS by Juraweiss and 13 g of product E according to the invention.

The results of the viscosity measurements as a function of the speed of flow according to DIN 125 at 20°C are set out in Tables Ia and Ib below.

TABLE Ia

		Prior art	Prior art	Invention	Prior art	Invention
COMPOSITION	TEST N°	1	2	3	4	5
	Plastisol without filler (in g)	75.00	75.00	75.00	75.00	75.00
	PCC (in g)	20.00	13.00	-	25.00	10.00
	Juraperle™ BS (in g)	5.00	-	-	-	-
	Product A (in g)	-	12.00	-	-	-
	Product B (in g)	-	-	25.00	-	-
	Product D (in g)	-	-	-	-	15.00
	Weight of mixture (in g)	100.00	100.00	100.00	100.00	100.00
RHEOLOGY	Viscosity mPa.s / Speed of flow s -1	Test N° 1	Test N° 2	Test N° 3	Test N° 4	Test N° 5
	20	18200	17700	15200	17600	17100
	40	10040	9650	8430	9900	9650
	60	7260	7100	6280	7200	7100
	80	5890	5730	5170	5850	5700
	100	5100	4880	4390	5000	4800
	120	4490	4380	3980	4450	4350
	140	4100	4020	3550	3990	3990
	160	3780	3650	3310	3650	3650
	180	3460	3400	3110	3400	3380
	200	3320	3220	2950	3190	3170
	220	3140	3030	2780	3110	3000
	240	3000	2870	2620	2890	2820
	260	2870	2760	2520	2740	2700
	280	2760	2680	2420	2610	2600
	300	2660	2570	2350	2490	2490

TABLE Ib

		Invention	Invention	Invention	Invention	Invention
COMPOSITION	Test N°	6	7	8	9	10
	Plastisol without filler (in g)	75.00	75.00	75.00	75.00	72.00
	PCC (in g)	10.00	13.00	10.00	10.00	10.00
	Juraperle™ BS (in g)	-	-	5.00	5.00	5.00
	Product C (in g)	15.00	-	-	-	-
	Product D (in g)	-	-	10.00	-	-
	Product E (in g)	-	12.00	-	10.00	13.00
	Weight of mixture (in g)	100.00	100.00	100.00	100.00	100.00
RHEOLOGY	Viscosity mPa.s / Speed of flow s^{-1}	Test N° 6	Test N° 7	Test N° 8	Test N° 9	Test N° 10
	20	12700	17300	13100	12500	17000
	40	7280	9450	7600	7160	9500
	60	5360	6950	5630	5250	7030
	80	4430	5600	4850	4330	5580
	100	3820	4830	4040	3780	4800
	120	3400	4230	3600	3300	4320
	140	3110	3950	2290	3000	3790
	160	2860	3550	3040	2770	3550
	180	2680	3260	2850	2590	3280
	200	2520	3110	2680	2480	3080
	220	2400	2910	2550	2320	2950
	240	2290	2790	2430	2200	2800
	260	2190	2670	2340	2130	2710
	280	2120	2580	2250	2050	2610
	300	2040	2490	2180	1970	2490

A reading of the table shows that the use of the calcium carbonate according to the invention makes it possible to regulate the rheological behaviour of the plastisol composition, even when 100% of the precipitated calcium carbonate has been replaced by the calcium carbonate according to the invention.

EXAMPLE 2:

This example relates to the study of the conventional mechanical properties conferred by product F according to the invention, that is to say a natural chalk crushed so as to obtain a specific surface area of 28 m^2/g, on mixtures based on plasticised PVC, in comparison with mixtures filled by means of natural calcium carbonates well known in the prior art.

For each of these tests, the following mixture was produced:

"SOLVIC 239 D" PVC sold by Solvay	100
Dioctyl phthalate (Jayflex™ DOP from Exxon)	50
Dibasic lead sulphate	1
Tribasic lead sulphate	2
Filler to be tested	80

Preparation of the composition:

The dry mixtures or "dry blends" were prepared in a "GUEDU"™ adiabatic mixer at 100°C for a period of 15 minutes. The mixtures were then gelled on cylinders at 150°C in a mixing mill. For all the mixtures, this operation was performed in 12 minutes.

Sheets of 90 x 90 x 2 mm were then moulded at 160°C after preheating of the blank for 3 minutes and pressurising for 2 minutes before cooling, using a compression press.

The test pieces necessary for determining the mechanical properties were cut from these sheets.

The different calcium carbonates tested were:

Test N° 11:

This test illustrates the prior art and uses a natural chalk, treated with 1% stearic acid, with a specific surface area of 6 m^2/g measured according to the BET method to ISO 4652 (product I).

Test N° 12:

This test illustrates the prior art and uses a natural chalk, treated with 1% stearic acid, with a specific surface area of 10 m^2/g measured according to the BET method to ISO 4652 (product J).

Test N° 13:

This test illustrates the invention and uses product F according to the invention.

The mechanical properties were assessed by means of dynamometric tests (tensile strength, breaking elongation, modulus 100%) carried out using the Instron™ equipment according to ISO 37, at a temperature 23°C and with a traction speed of 10 cm/min.

The ASTM-C tear strength was for its part determined according to the method of ISO R-34 and Shore C hardness according to the method of ISO 868.

The results of these measurements of mechanical properties are set out in Table II below.

TABLE II

Test N°	Prior art 11	Prior art 12	Invention 13
Tensile strength (daN/cm^2)	128	130	136
Breaking elongation (%)	300	260	172
Modulus 100% (daN/cm^2)	83	106	127
ASTM-C tear strength (daN/cm)	47	56	58
Shore C hardness at 15 s (in daN/cm)	60	64	74

It can be seen that the mechanical properties obtained are excellent and are superior to those obtained with natural calcium carbonates which are crushed but which have a specific surface area outside the scope of the invention.

The invention therefore makes it possible to optimise the formulations according to the mechanical property to be favoured.

EXAMPLE 3:

This example relates to the use of calcium carbonates as a rheology regulator for the preparation of elastomer based on natural or synthetic rubber.

It was sought in this example to assess the effect of the specific surface area of a crushed natural chalk according to the invention on the properties of mixtures based on natural and synthetic rubbers, in comparison with a precipitated calcium carbonate of the prior art.

To do this, for each of Tests 14 and 15, the following mixture was produced:

Natural rubber (smoked sheet quality RSS 1)	100
SBR rubber (styrene-butadiene, Cariflex™ 1502 from Shell)	40
Zinc oxide (snow quality) from Vieille Montagne	5
Stearic acid	2
Sulphur	1.5
N-cyclohexyl 2 benzothiazyl sulphenamide (Vulcafor™ CBS from Vulnax)	1
Tetramethylthiuram disulphide (Vulkacit™ DTMT from Bayer)	0.5
Calcium carbonate to be tested	100

Test N° 14:

This test illustrates the prior art and uses a precipitated calcium carbonate sold by Solvay under the name Socal U1S2 (product K).

Test N° 15:

This test illustrates the invention and uses product F according to the invention.

These two tests were carried out as follows:

A pure gum master-batch was prepared, by the successive incorporation of the various ingredients, except for calcium carbonate, according to the normal technique of experts, on a mixing mill regulated for temperature, by mixing for 10 minutes (friction I/I,4) at 60°C.

From this master-batch two samples were taken in which the calcium carbonates to be tested had been incorporated, by mixing at 60°C for 12 minutes.

After determination of the vulcanisation optima at 155°C using a Monsanto flow meter, sheets were moulded and vulcanised to this optimum in order to effect the measurement of the mechanical properties according to the same operating method as in the previous tests.

The results of the mechanical properties are set out in Table III below.

TABLE III

	Prior art	Invention
Test N°	14	15
Vulcanisation optimum at 155°C	5 min. 15 s	5 min.
Tensile strength daN/cm^2	119	109
Modulus 300% daN/cm^2	41	39
Elongation %	500	485
Tear strength ASTM-C daN/cm	23	26
Shore A hardness (15 s)	61	61

It can be seen that the product according to the invention reduces the implementation time (the vulcanisation optimum) as well as the tear strength properties.

Likewise Tests 16, 17 and 18 were carried out using the following formulation:

SBR rubber (styrene-butadiene, Cariflex™ 1502 from Shell)	40
Natural rubber (smoked sheet quality RSS 1)	60
Zinc oxide (snow quality) from Vieille Montagne	5
Stearic acid	2
Sulphur	2
N-cyclohexyl 2 benzothiazyl sulphenamide (Vulcafor™ CBS from Vulnax)	0.9
Diorthotolylguanidine accelerator (Vulkafor™ DOTG from Vulnax)	0.3
Calcium carbonate to be tested	100

Test N° 16:

This test illustrates the prior art and uses a precipitated calcium carbonate sold by Solvay under the name Socal U1S2 (product K).

Test N° 17:

This test illustrates the prior art and uses a calcium carbonate of the chalk type, crushed by the wet method and dried, treated, with a mean diameter of 1.4 micrometres measured by means of the Sedigraph 5100 from Micromeritics and with a specific surface area of 10 m^2/g measured according to the BET method to ISO 4652 (product J).

Test N° 18:

This test illustrates the invention and uses product G according to the invention.

From this master batch manufactured with the same operating method as in the previous test, three samples were taken in which the calcium carbonates to be tested had been incorporated, by mixing at 60°C for 12 minutes.

After determination of the vulcanisation optima, sheets were moulded and vulcanised to this optimum in order to effect a measurement of the mechanical properties according to the same operating method as for the previous tests.

The results of the mechanical properties are set out in Table IV below.

TABLE IV

	Prior art	Prior art	Invention
Test N°	16	17	18
Vulcanisation optimum at 150°C	23 min. 30 s	16 min. 45 s	11 min.
Tensile strength (daN/cm^2)	122	132	132
Modulus 300% (daN/cm^2)	16.5	20	20
Elongation %	695	715	695
ASTM-C tear strength (daN/cm)	23	28	28
Shore A hardness (15 s) in (daN/cm)	51	50	50

It can be seen that the product according to the invention improves the implementation time (the vulcanisation optimum) and the majority of the mechanical properties.

EXAMPLE 4:

A second series of tests (Tests 19 and 20) were carried out in a natural rubber 40 pcr filled with a mineral filler with the basic formula:

Natural rubber (smoked sheet quality RSS 1)	100
Coumarone resin 60/70	5.6
Rosin	3
Zinc oxide (snow quality) from Vieille Montagne	40
Stearic acid	0.5
Oil	4.3
Benzothiazyl disulphide accelerator (Vulcafor™ MBTS from Vulnax)	1
Diphenylguanidine accclerator (Vulcafor™ DPG from Vulnax)	0.36

The calcium carbonates to be tested were:

Test N° 19:

A precipitated calcium carbonate (product K) for this test, which illustrates the prior art.

Test N° 20:

A natural calcium carbonate according to the invention (product G) for this test, which illustrates the invention.

The results of the mechanical properties, measured with the same operating method as for the following tests, are set out in Table V below.

TABLE V

	Prior art	Invention
Test N°	19	20
Vulcanisation optimum at 150°C	7 min. 15 s	8 min. 15 s
Tensile strength (daN/cm^2)	246	246
Modulus 300% (daN/cm^2)	32	31
Elongation %	710	710
Shore A hardness (15 s) in (daN/cm)	45	46,5

It can be seen that the product according to the invention makes it possible to obtain equivalent mechanical property results, even by completely replacing the precipitated calcium carbonate with a natural calcium carbonate.

CLAIMS

1. A rheology regulator, characterised in that it is a case of a natural calcium carbonate, crushed to a high degree of fineness, with a specific surface area of around 14 to 30 m^2/g, preferably around 16 to 24 m^2/g and highly preferentially around 20 m^2/g, measured according to the BET method to ISO 4652.

2. A rheology regulator according to Claim 1, characterised in that it is a case of a natural calcium carbonate, crushed to a high degree of fineness, with a specific surface area of 14.4 m^2/g, measured according to the BET method to ISO 4652.

3. A rheology regulator according to Claim 1, characterised in that it is a case of a natural calcium carbonate, crushed to a high degree of fineness, with a specific surface area of 16 m^2/g, measured according to the BET method to ISO 4652.

4. A rheology regulator according to Claim 1, characterised in that it is a case of a natural calcium carbonate, crushed to a high degree of fineness, with a specific surface area of 16.5 m^2/g, measured according to the BET method to ISO 4652.

5. A rheology regulator according to Claim 1, characterised in that it is a case of a natural calcium carbonate, crushed to a high degree of fineness, with a specific surface area of 22 m^2/g, measured according to the BET method to ISO 4652.

6. A rheology regulator according to Claim 1, characterised in that it is a case of a natural calcium carbonate, crushed to a high degree of fineness, with a specific surface area of 28 m^2/g, measured according to the BET method to ISO 4652.

7. A rheology regulator according to any one of Claims 1 to 6, characterised in that it is a case of a natural calcium carbonate treated by means of at least one fatty acid containing 10 to 24 atoms of carbon or its salt chosen from amongst the salts of calcium, magnesium, zinc or a mixture thereof and more particularly using stearic acid or its calcium salt in a proportion of around 0.01% to 5% by weight.

8. A rheology regulator according to Claim 7, characterised in that it is a case of a natural calcium carbonate treated by means of at least one fatty acid containing 10 to 24 atoms of carbon or its salt chosen from amongst the salts of calcium, magnesium, zinc or a mixture thereof and more particularly using stearic acid or its calcium salt in a proportion of around 1% to 4% by weight.

9. A rheology regulator according to any one of Claims 1 to 8, characterised in that it has an oil absorption which is greater than 16 measured according to ISO 787-V (Rub-out method).

10. Use of a rheology regulator according to any one of Claims 1 to 9 for the preparation of sealants, adhesives or plastisols.

11. Use of a rheology regulator according to any one of Claims 1 to 9 for the preparation of rubbers.

12. Use as a rheology regulator, of dispersions or suspensions, in an organic medium, of a natural calcium carbonate crushed to a high degree of fineness according to any one of Claims 1 to 9 for the preparation of sealants or coatings, adhesives or plastisols.

13. Use as a rheology regulator, of dispersions or suspensions, in an organic medium, of a natural calcium carbonate crushed to a high degree of fineness according to any one of Claims 1 to 9, for the preparation of rubbers.

14. A plastisol, characterised in that it comprises a rheology regulator according to any one of Claims 1 to 9.

15. A rubber, characterised in that it comprises a rheology regulator according to any one of Claims 1 to 9.

16. A sealant or coating or adhesive characterised in that it comprises a rheology regulator according to any one of Claims 1 to 9.

17. A sealant or coating or adhesive according to Claim 16, characterised in that it comprises in addition a polyurethane with terminal silane groups and a plasticiser of the phthalate type.

18. A sealant or coating or adhesive according to either one of Claims 16 and 17, characterised in that it comprises in addition one or more additives chosen from amongst smoked silica as a thixotropic agent, a bleaching agent such as TiO_2,

UV stabilisers, adhesion promoter, a catalyst such as dibutyltin dilaurate, and dehydrating agents such as a silane.

ABSTRACT

The invention relates to the selection of a natural calcium carbonate crushed to a high degree of fineness, possibly treated with one or more fatty acids or one or more of their salts or mixtures thereof, and its use as a rheology regulator for polymeric compositions.

The specific surface area is 14 to 30 m^2/g measured according to the BET method to ISO 4652, and the oil absorption is greater than 16 measured according to ISO 787-V (Rub-out method).

The conditions of mixing of the constituents and manufacture are improved, as well as the properties of the final product.

205738US

Declaration and Power of Attorney for Patent Application
Déclaration et Pouvoirs pour Demande de Brevet

French Language Declaration

En tant l'inventeur nommé ci-après, je déclare par le présent acte que:

As a below named inventor, I hereby declare that:

Mon domicile, mon adresse postale et ma nationalité sont ceux figurant ci-dessous à côté de mon nom.

My residence, mailing address and citizenship are as stated next to my name.

Je crois être le premier inventeur original et unique (si un seul nom est mentionné ci-dessous), ou l'un des premiers co-inventeurs originaux (si plusieurs noms sont mentionnés ci-dessous) de l'objet revendiqué, pour lequel une demande de brevet a été déposée concernant l'invention intitulée

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled.

NOVEL RHEOLOGY REGULATORS SUCH AS GROUND NATURAL CALCIUM CARBONATES OPTIONALLY TREATED WITH A FATTY ACID OR SALT AND THEIR USE (AS AMENDED)

et dont la description est fournie ci-joint à moins

the specification of which

☐ ci-joint

☐ a été déposée le _____

sous le numéro de demande des Etats-Unis ou le numéro de demande international PCT

_____ et modifiée le

_____ (le cas échéant).

☐ is attached hereto.

☒ was filed on April 9, 2001

as United States Application Number or PCT International Application Number

09/806,473 and was amended on

_____ (if applicable)

Je déclare par le présent acte avoir passé en revue et compris le contenu de la description ci-dessus, revendications comprises, telles que modifiées par toute modification dont il aura été fait référence ci-dessus.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

Je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56.

French Language Declaration

Je revendique par le présent acte avoir la priorité étrangère, en vertu du Titre 35, § 119(a)-(d) ou § 365(b) du Code des Etats-Unis, sur toute demande étrangère de brevet ou certificat d'inventeur ou, en vertu du Titre 35, § 365(a) du même Code, sur toute demande internationale PCT désignant au moins un pays autre que les Etats-Unis et figurant ci-dessous et, en cochant la case, j'ai aussi indiqué ci-dessous toute demande étrangère de brevet, tout certificat d'inventeur ou toute demande internationale PCT ayant une date de dépôt précédant celle de la demande à propos de laquelle une priorité est revendiquée.

I hereby claim foreign priority under Title 35, United States Code, § 119 (a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application(s)
Demande(s) de brevet anterieure(s) dans un autre pays.

(Number) (Numéro)	(Country) (Pays)	(Day/Month/Year Filed) (Jour/Mois/Anné de dépôt)	Priority Claimed Droit de priorité Revendiqué	
98/12714	FRANCE	07 OCTOBER 1998	☒ Yes Oui	☐ No Non
			☐ Yes Oui	☐ No Non

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 119(e) du Code des Etats-Unis, de toute demande de brevet provisoire effectuée aux Etats-Unis et figurant ci-dessous.

I hereby claim the benefit under Title 35, United States Code, §119(e) of any United States provisional application(s) listed below.

(Application No.) (Nº de demande)	(Filing Date) (Date de dépôt)

Je revendique par le présent acte tout bénéfice, en vertu du Titre 35, § 120 du Code des Etats-Unis, de toute demande de brevet effectuée aux Etats-Unis, ou en vertu du Titre 35, § 365(c) du même Code, de toute demande internationale PCT désignant les Etats-Unis et figurant ci-dessous et, dans la mesure où l'objet de chacune des revendications de cette demande de brevet n'est pas divulgué dans la demande antérieure américaine ou internationale PCT, en vertu des dispositions du premier paragraphe du Titre 35, § 112 du Code des Etats-Unis, je reconnais devoir divulguer toute information pertinente à la brevetabilité, comme défini dans le Titre 37, § 1.56 du Code fédéral des réglementations, dont j'ai pu disposer entre la date de dépôt de la demande antérieure et la date de dépôt de la demande nationale ou internationale PCT de la présente demande:

I hereby claim the benefit under Title 35, United States Code, § 120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of Title 35, United States Code, § 112, I acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations, § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

(Application No.) (Nº de demande)	(Filing Date) (Date de dépôt)	(Status: Patented, Pending, Abandoned) (Statut : breveté, en cours d'examen, abandonné)
PCT/IB99/01625	01 OCTOBER 1999	

Je déclare par le présent acte que toute déclaration ci-incluse est, à ma connaissance, véridique et que toute déclaration formulée à partir de renseignements ou de suppositions est tenue pour véridique; et de plus, que toutes ces déclarations ont été formulées en sachant que toute fausse déclaration volontaire ou son équivalent est passible d'une amende ou d' une incarcération, ou des deux, en vertu de la § 1001 du Titre 18 du Code des Etats-Unis, et que de telles déclarations volontairement fausses risquent de compromettre la validité de la demande de brevet ou du brevet délivré à partir de celle-ci.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

French Language Declaration

POUVOIRS: En tant que l'inventeur cité, je désigne par la présente l'(les) avocat(s) suivant(s) pour qu'ils poursuive(nt) la procédure de cette demande de brevet et traite(nt) toute affaire s'y rapportant avec l'Office des brevets et des marquees: *(mentionner le nom et le numéro d'enregistrement).*

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith: (list name and registration number)

022850

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(Fournir les mêmes renseignements et la signature du troisième co-inventeur et de tout co-inventeur supplémentaire.)

(Supply similar information and signature for third and subsequent joint inventors.)

French Language Declaration

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Signature de l'inventeur Date	Fourth inventor's signature Date June 6, 2002
Domicile	Residence Henry Loman residence :Obersumpfstrasse 38, CH-5745 Safenwil, SWITZERLAND; Marion LOMAN-OONK residence Vriezelaan 11, NL-7602 JJ Almelo, NETHERLANDS
Nationalité	Citizenship of both Henry LOMAN and Marion LOMAN-OONK is NETHERLANDS
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Nom complet du cinquième co-inventeur, le cas echeant	Full name of fifth joint inventor, If any
Signature de l'inventeur Date	Fifth inventor's signature Date
Domicile	Residence
Nationalité	Citizenship
Adresse Postale	Mailing Address

Nom complet du sixième co-inventeur, le cas echeant	Full name of sixth joint inventor, If any
Signature de l'inventeur Date	Sixth inventor's signature Date
Domicile	Residence
Nationalité	Citizenship
Adresse Postale	Mailing Address

(Fournir les mêmes renseignements et la signature du septième co-inventeur et de tout co-inventeur supplémentaire.)

(Supply similar information and signature for seventh and subsequent joint inventors.)